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# Operator's Manual for the Compact X-Ray Analyzer

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Material Sciences Division

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NAVAL RESEARCH LABORATORY  
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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number)<br>A low cost, compact wavelength dispersion x-ray fluorescence analyzer has been built by the Naval Research Laboratory for the Environmental Protection Agency. This report documents the recommended method of operation of this instrument and includes diagrams of the special components fabricated for it. |                                                                                                                                                                                |                                             |

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Operator's Manual for the  
Compact X-Ray Analyzer

## INTRODUCTION

The Compact X-Ray Analyzer (CXA) built at the Naval Research Laboratory (NRL) for the Environmental Protection Agency (EPA) is a low power, low cost wavelength dispersive x-ray fluorescence instrument designed to measure particulate samples collected from emission sources. It uses state-of-the-art components where they are commercially available and specially fabricated parts elsewhere. This manual describes the instrument and its operation in sufficient detail for routine use. Details of the construction and testing of the CXA can be found in the Final Report. (1)

## OVERALL DESIGN

The CXA is a self-contained instrument, the single cabinet housing the x-ray generator, the crystal spectrometer, and the counting and readout electronics. A block diagram is shown in Figure 1, and photographs in Figures 2 and 3. Table 1 lists the commercial components and the parts fabricated at NRL.

## COMPONENTS

**Regulator:** The Sola regulator powers the entire instrument, providing a stable 120 volts A. C. for an input of 95 to 130 volt, 60 Hertz, single phase line voltage.

**X-Ray Generator:** The x-ray generator consists of a variac-controlled high-voltage supply, a variac-controlled filament supply, a kilovolt meter, a milliamp meter, an x-ray tube, and an air-cooled, oil insulated tube housing. The high-voltage supply is rated at + 40 kV (c.p.), 4 mA. Input from the variac is connected to pins 1 and 3 of the 8 pin connector; pin 7 is grounded. The output cable from the high-voltage supply is fed into the oil-filled tube housing through a sealing gland. Figure 4 is an exploded sketch of the x-ray tube and housing. The shield of the high-voltage cable is connected to ground through a 5 mA meter which measures the x-ray tube current. Inside the tube housing is a 1000 megohm resistor stack to which the front

Note: Manuscript submitted September 9, 1975.

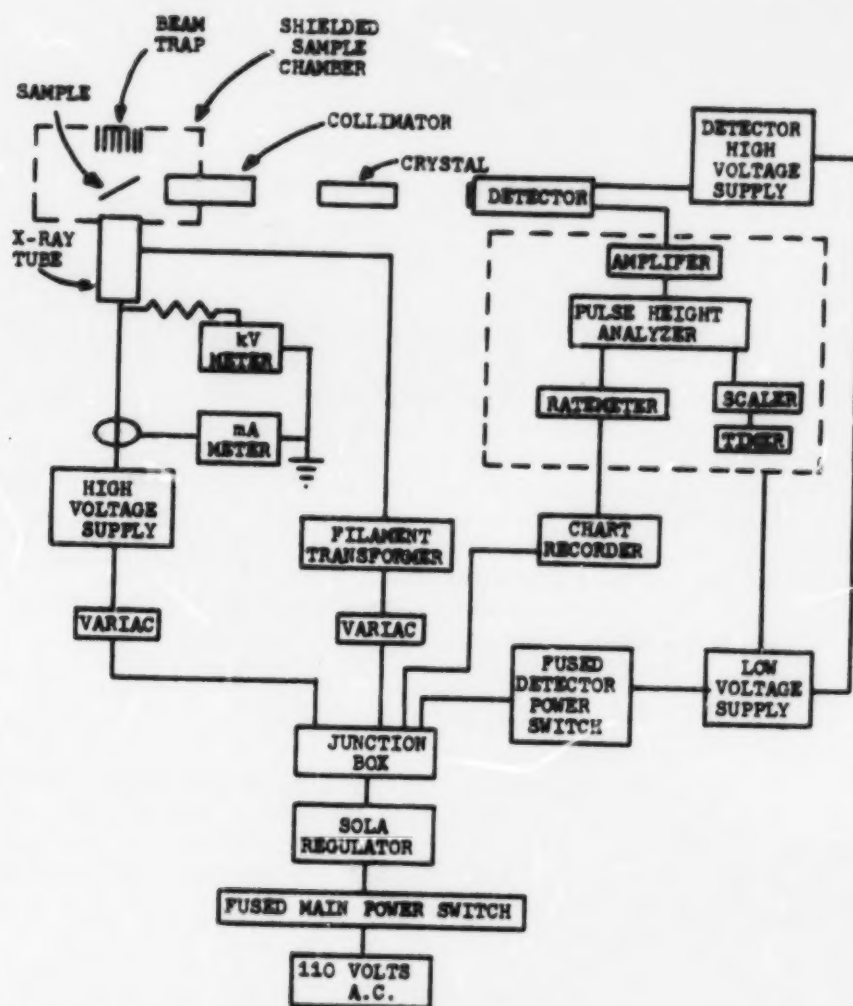


Fig. 1 Block diagram of the Compact X-Ray Analyzer.

panel voltmeter is connected. This meter, although labelled "micro-amps," reads directly in kilovolts; it has been calibrated with an electrostatic voltmeter, as shown in Figure 5. The electrostatic voltmeter was limited to 30 kV, so the upper half of the range was measured half-way on the resistor stack. The filament of the x-ray tube is heated by a variac-controlled 6 volt, 5 ampere transformer. The filament connections are on the anode end of the x-ray tube. Therefore the filament leads are brought in through the sample chamber, as shown in Figures 3 and 4. The NRL-built x-ray tube housing is machined from aluminum with cooling fins, past which air is blown by a fan. The housing is filled with conventional transformer oil; temperature rise during operation causes expansion so that the oil level

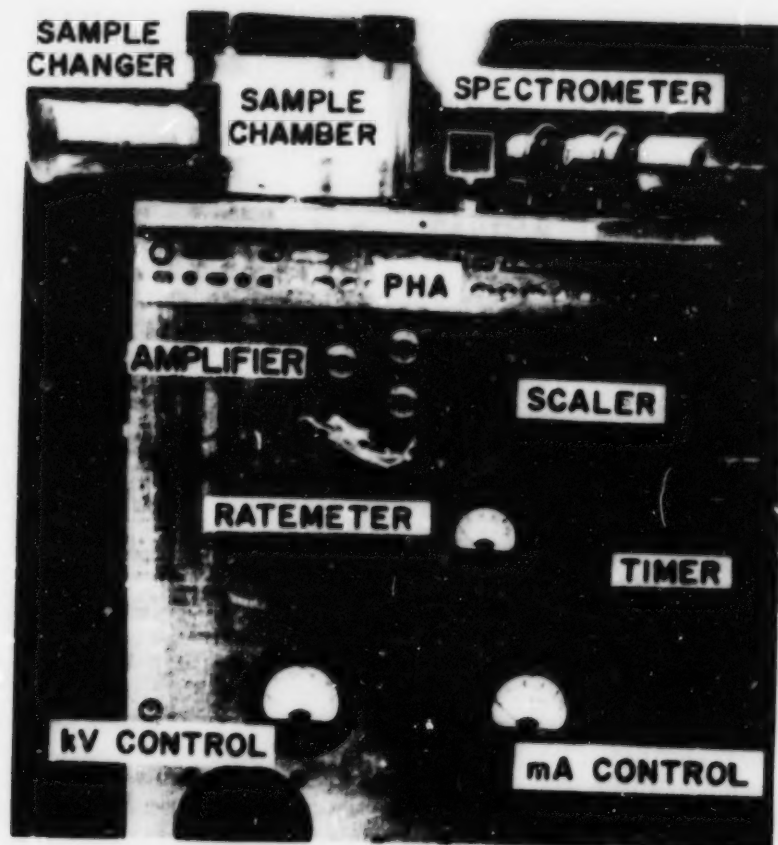


Fig. 2 Photograph of the Compact X-Ray Analyzer.

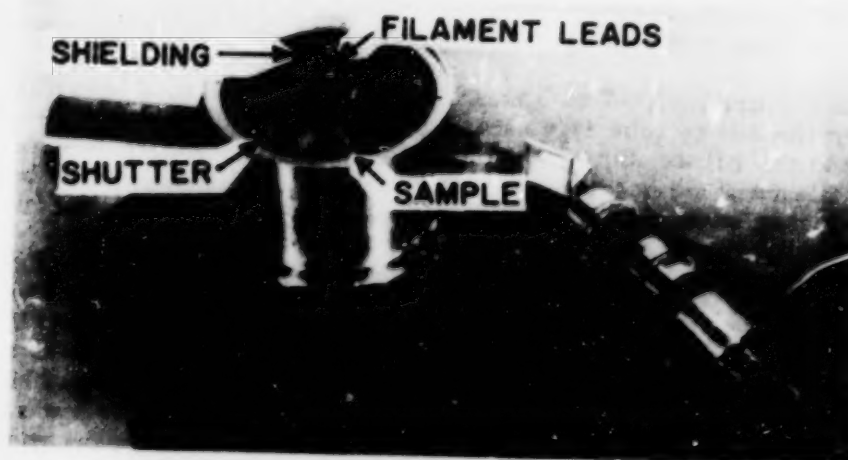


Fig. 3 Photograph of the spectrometer with the sample chamber open.

TABLE I

## Components for Single Element Analyzer

Purchased Commercially

Regulator  
 X-Ray Tube H.V. Supply  
 X-Ray Tube  
 ".005 x 4" Collimator  
 Scintillation Detector  
 Detector H.V. Supply  
 Recorder  
 Electronic Power Supply

Manufacturer and Model Number

Sola 23-22-150  
 Universal-Voltronics BPE-40-4  
 Machlett EG-50 HW  
 Philips 19011200  
 Harshaw SHG  
 Venus Scientific K-15  
 Rustrak 288  
 Sorensen MMD-12.120

Fabricated at NRL

Crystal (LiF)  
 Filament Supply  
 X-Ray Tube Housing  
 and Sample Chamber  
 Spectrometer, including  
 crystal and detector mounts  
 Counting Electronics:  
 Amp., PHA, Ratemeter  
 Scaler, Timer, etc.  
 Miscellaneous Cables  
 connectors and electronics comp.  
 Cabinet to house components  
 fabricated at NRL

Cleaved, abraded and etched,  
 from NRL stock  
 6.3 VCT Transformer, 1.5 Amp.  
 Variac and miscellaneous small  
 parts  
 Manufactured in NRL Shops  
 Gears plus machine work in  
 NRL Shops  
 Some circuitry designed in NRL  
 Electronic Shop, other circuitry  
 standard

is fairly critical. The housing should be filled to a level such that when the x-ray tube is operating at its normal power (80 watts-40 kV, 2 MA) the oil expands to provide the cooling medium in contact with the filament connections near the window end of the tube, but not so much that it overflows the housing. There is a fill-level mark on the lead shielding lining the housing.

Specimen Chamber: The specimen chamber is also a machined aluminum component which fits over the tube housing and is fitted with a sample changing mechanism, a collimator and a removable top which centers a beam trap made of lead-foil tubes behind the sample above the tube window. Like the tube housing, the sample chamber is lined with lead to make it radiation safe. A lead shutter is spring-loaded so that radiation does not escape when the sample changing mechanism is withdrawn from the chamber.

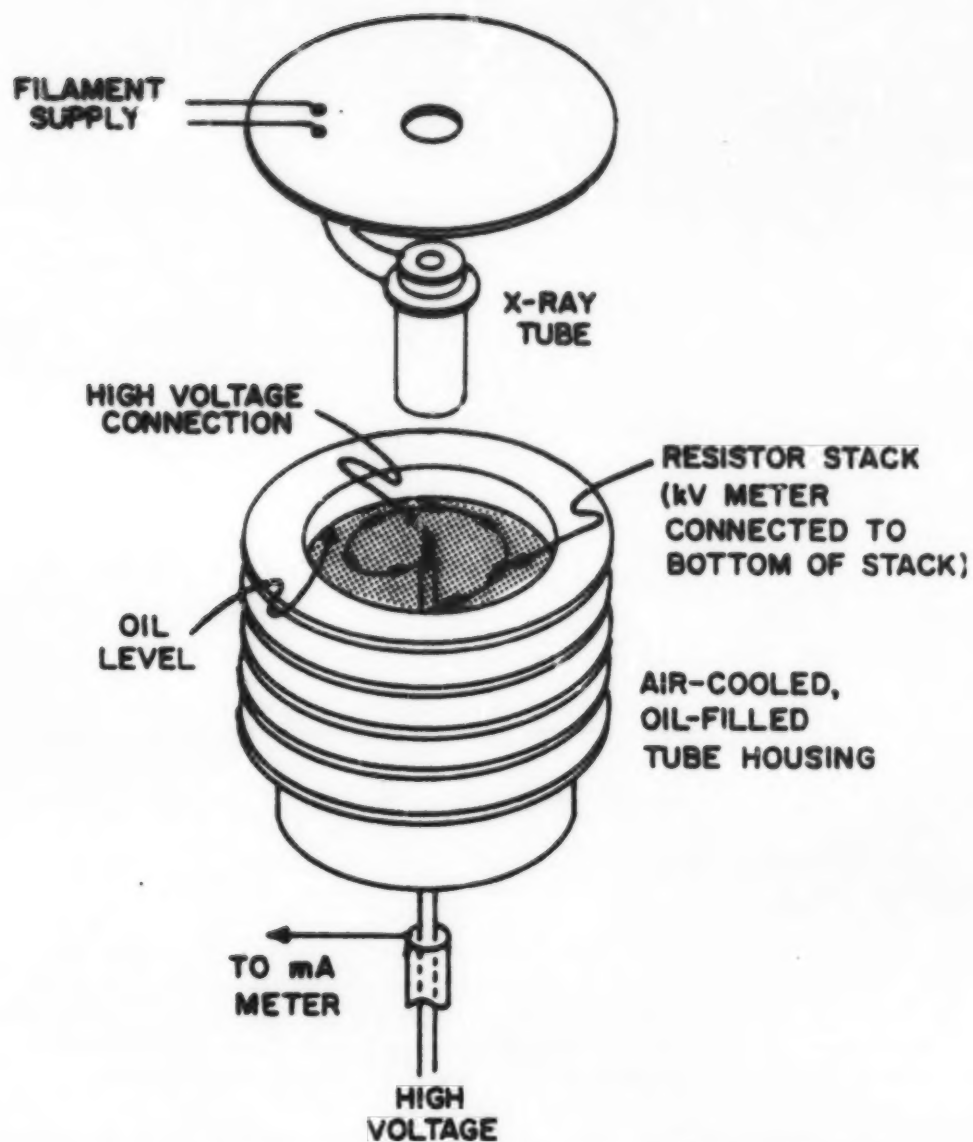


Fig. 4 Exploded sketch of the x-ray tube and housing.

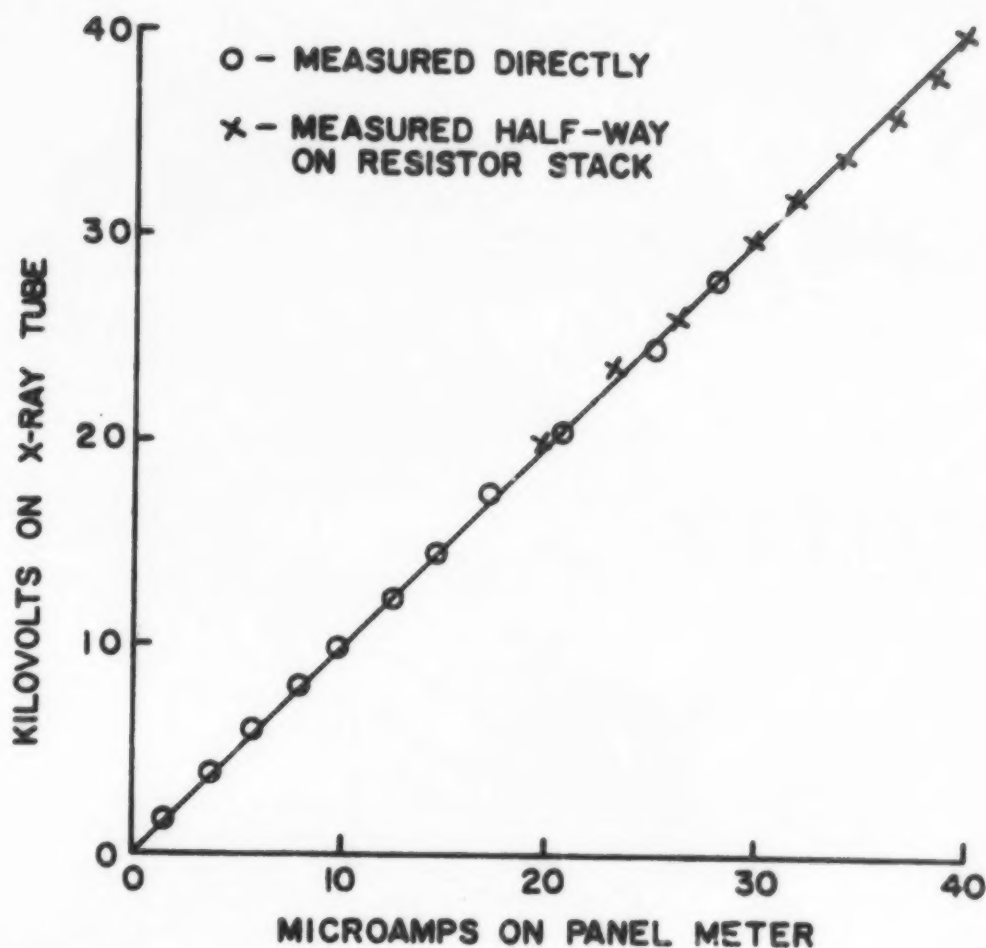


Fig. 5 Electrostatic calibration of x-ray tube power supply.

**WARNING:** When the top of the specimen chamber is to be removed for any purpose the instrument should be disconnected from the 110 volt A.C. power source, so as to remove any possibility of the x-rays being turned on without the shielding in place. None of the lead shielding should ever be removed from any part of the instrument.

**X-Ray Spectrometer:** A plan view of the spectrometer is shown in Figure 6. The spectrometer provides  $\theta$ - $2\theta$  coupling between the crystal and detector by a planetary-gear arrangement as shown schematically in Figure 7. A photograph of the arrangement is shown in Figure 8. The crystal is a 1" x 2" x 1/8" abraded and etched (200) LiF. The crystal is attached to the mount by double coated pressure sensitive tape.

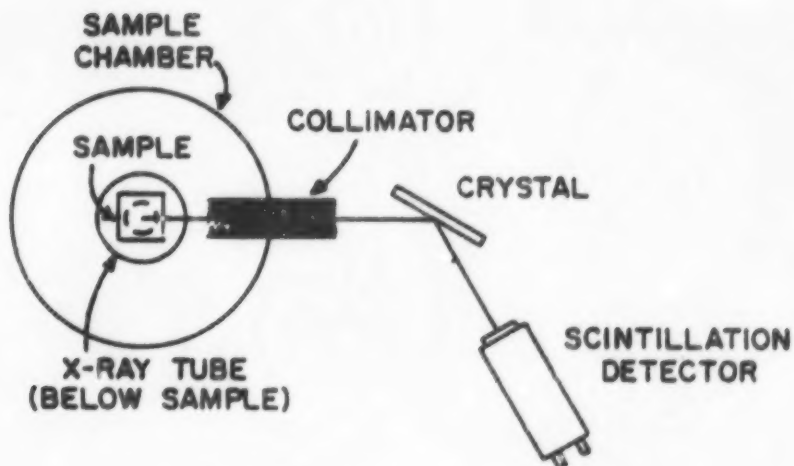


Fig. 6 Plan view of spectrometer.

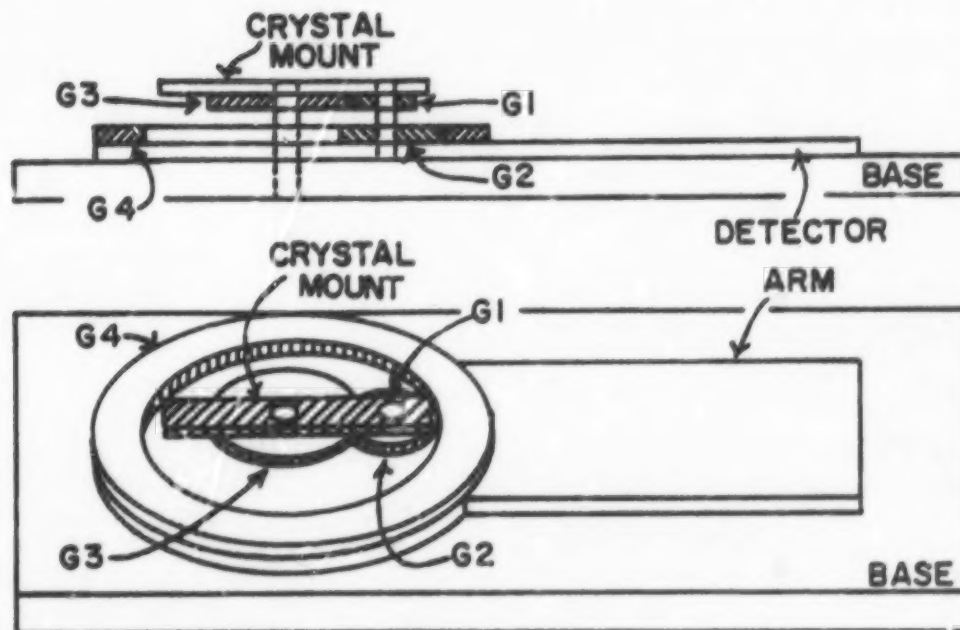


Fig. 7. Sketch of planetary gear arrangement.  
 (Gear ratios  $G1:G2:G3:G4 = 1:2:3:6$   
 G1 and G2 pinned to their shaft  
 G3 and Base pinned to their shaft  
 G4 pinned to Detector Arm.)

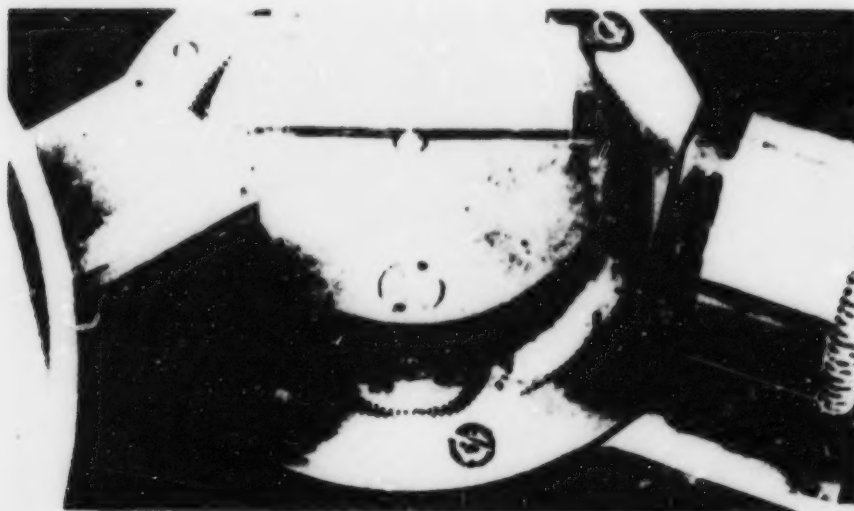


Fig. 8 Photograph of planetary gear arrangement.

Counting and Readout Electronics: The electronics package consists of a detector high voltage supply, an amplifier, a pulse height analyzer, a ratemeter, a scaler, and a timer, all powered by a  $\pm 12$  volt D.C. supply. With the exception of the detector high voltage supply, all of the electronics were built at NRL; their circuit diagrams are collated at the end of this report. The detector high voltage supply, as listed in Table I, is a commercial component, simply consisting of a multivibrator and a transformer; it should look something like circuit 6. The ratemeter circuit is connected to the count rate meter through a normally-closed telephone jack. If a recorder is desired it is plugged into the jack and the rate meter is automatically disconnected.

### OPERATION

Operation of the instrument can best be described by illustrating the steps for measuring the sensitivity and detection limit for a filter paper standard (or any other "thin" standard):

- 1.) With kV and mA variacs at zero, turn on main power switch. Check to assure fan is operating.
- 2.) Set voltage and current on x-ray tube. Normal setting is 40 kV, 2 mA.
- 3.) Turn on detector power switch and allow instrument to warm up for 15 to 30 minutes.
- 4.) Mount a relatively high concentration standard ( $\geq 50 \mu\text{g}/\text{cm}^2$ ) of the element of interest on the sample holder and insert it into the chamber.

5.) Set detector arm at the appropriate  $2\theta$  angle, from a set of tables (e.g. reference 2), or by solving the Bragg equation using  $2d$  for (200) LiF as  $4.027 \text{ \AA}$ .

6.) With the amplifier gain set high (9.90), integral PHA and low baseline (0.50 or the minimum value which eliminates electronic noise), and with the scaler-ratemeter switch in the ratemeter position, fine tune the  $2\theta$  angle to produce a maximum count rate. (The ratemeter may have to be zeroed with a lead shutter over the outer end of the collimator.)

7.) Set the amplifier gain and the pulse height analyzer parameters according to the wavelength being measured.

8.) Change ratemeter-scaler switch to scaler, set 10 or 100 seconds on the timer, press the reset button and activate the count switch. At end of counting period, read scaler.

9.) Repeat 8.) with a blank substrate inserted in sample position to measure background.

10.) From 8.) and 9.), determine sensitivity [slope of the linear calibration curve in  $(\text{c/s})/(\mu\text{g}/\text{cm}^2)$ ] and detection limit in  $\mu\text{g}/\text{cm}^2$ .

11.) Repeat 8.) and 9.) with unknown sample and appropriate substrate. Calculate concentration of unknown.

12.) For cases where the calibration curve is not linear, e.g. bulk samples, more than a single standard will be required to establish the calibration.

The elements which can be measured by this instrument include atomic number ranges 23 to 48 (V to Cd) by using their K lines and from 59 (Pr) upwards using L lines. At the short wavelength end of the range, the K emission from Ru, Rh, Pd, Ag, and Cd, a special adaptation is required. The crystal is not long enough to intercept the entire x-ray beam transmitted by the collimator at the low Bragg angles required. Optimum geometry requires that masks be fitted over the inner end of the collimator and the detector window confining the size of the beam to that which will be intercepted by the crystal, as illustrated in Figure 9.

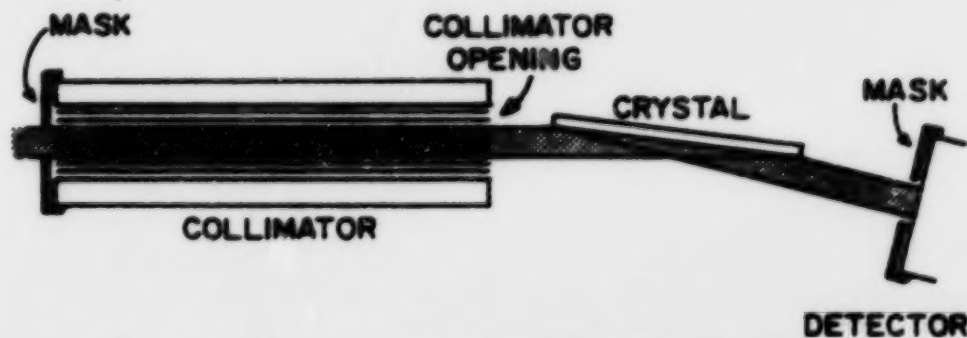
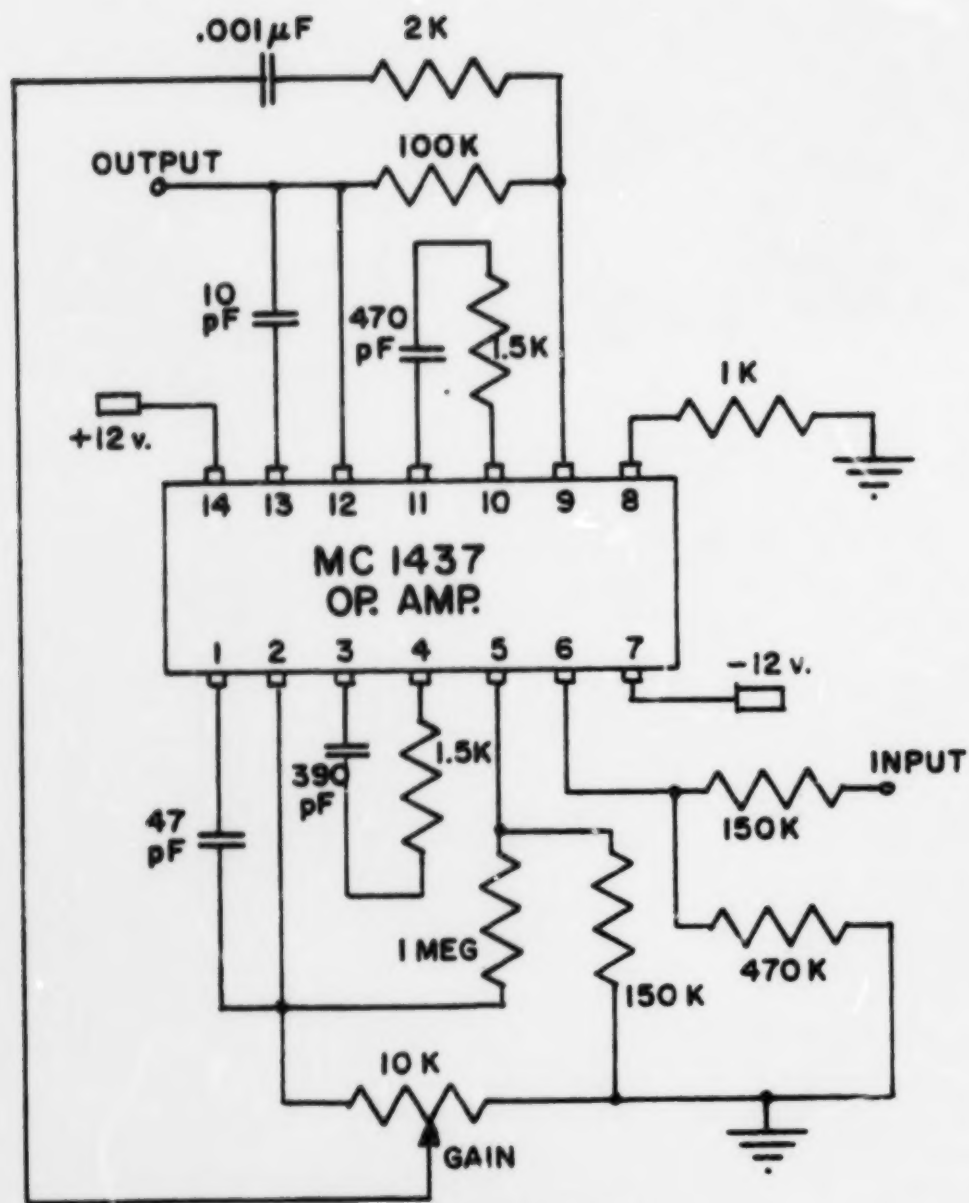


Fig. 9 Masked collimator and detector for short wavelength measurement. (Mask sizes used for Cd K $\alpha$  are:  $1/4''$  on the collimator and  $3/8''$  on the detector.)

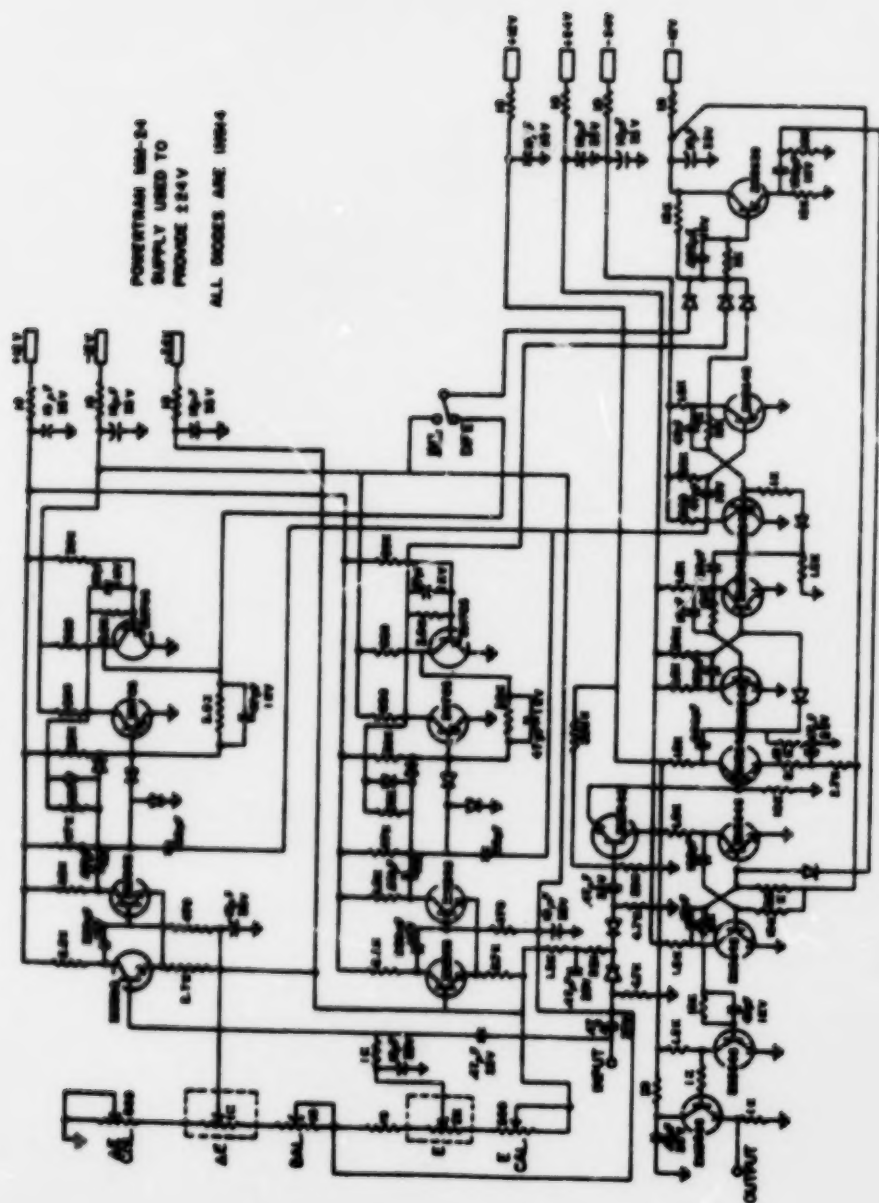
An instrument intended to analyze specifically for one of these elements probably should use a (220) LiF crystal for better dispersion and should be equipped with a 50 kV power supply for more efficient excitation.

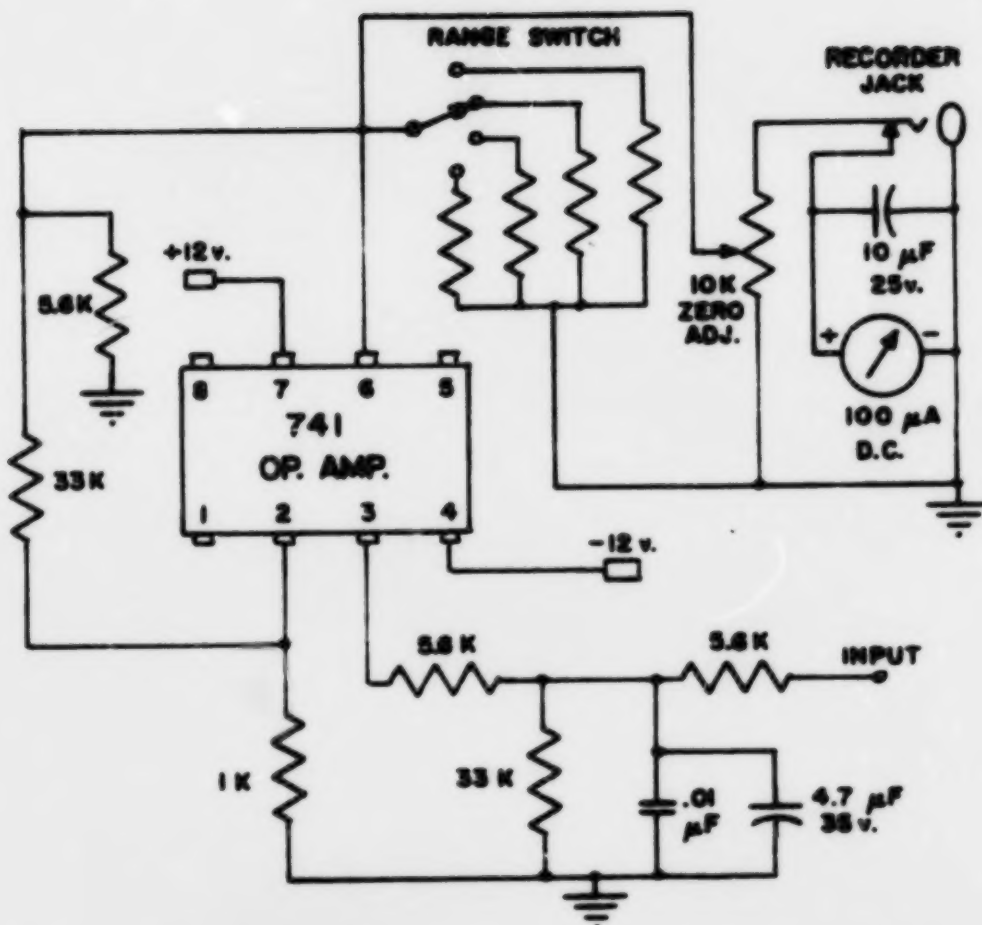
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- (1) "Low Cost Compact X-Ray Fluorescence Analyzer for On-Site Measurements of Single Elements in Source Emissions," L. S. Birks and J. V. Gilfrich, EPA-600/4-75-002, July 1975, and NRL Report 7926, August 26, 1975. *AD 801-284*
- (2) "Angular Positions of X-Ray Emission Lines of the Elements for Common Analyzing Crystals," Martin Berman and Sabri Ergun, Bureau of Mines Information Circular 8400, 1969.

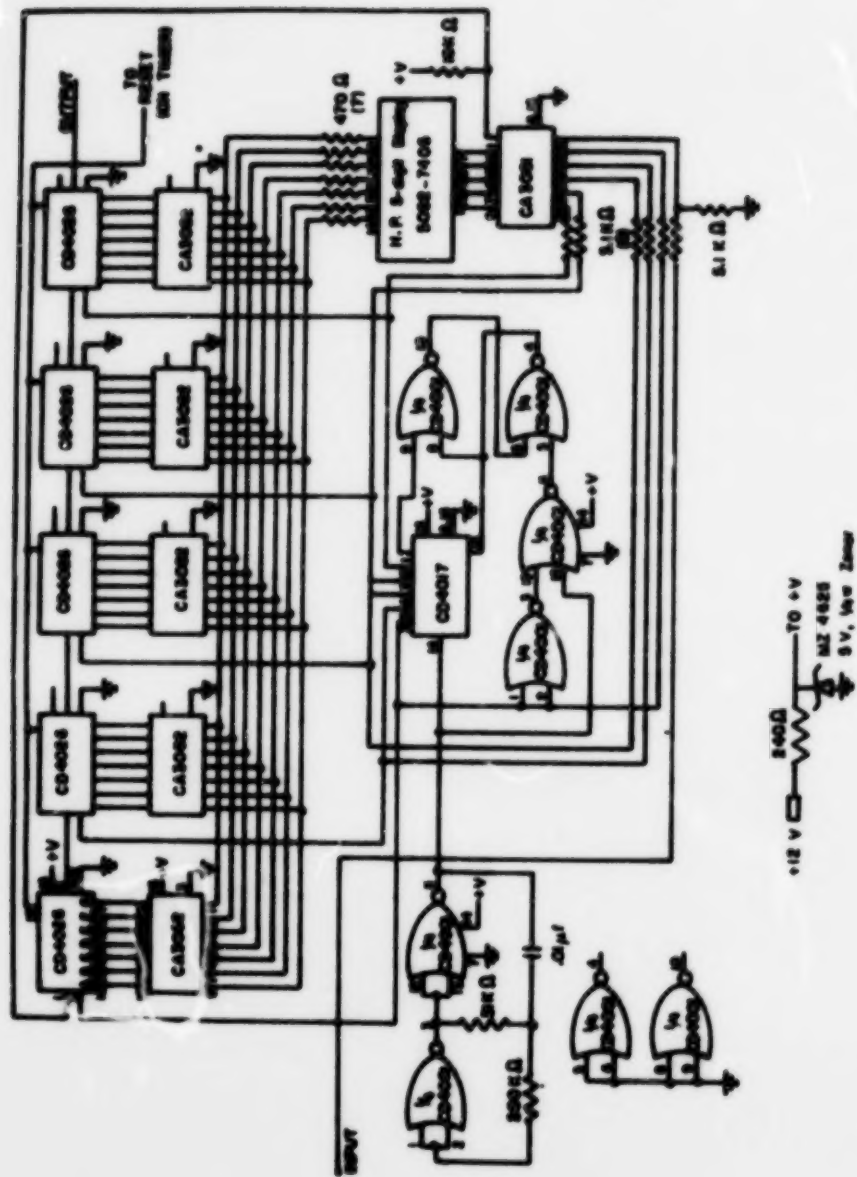


Circuit 1 Amplifier

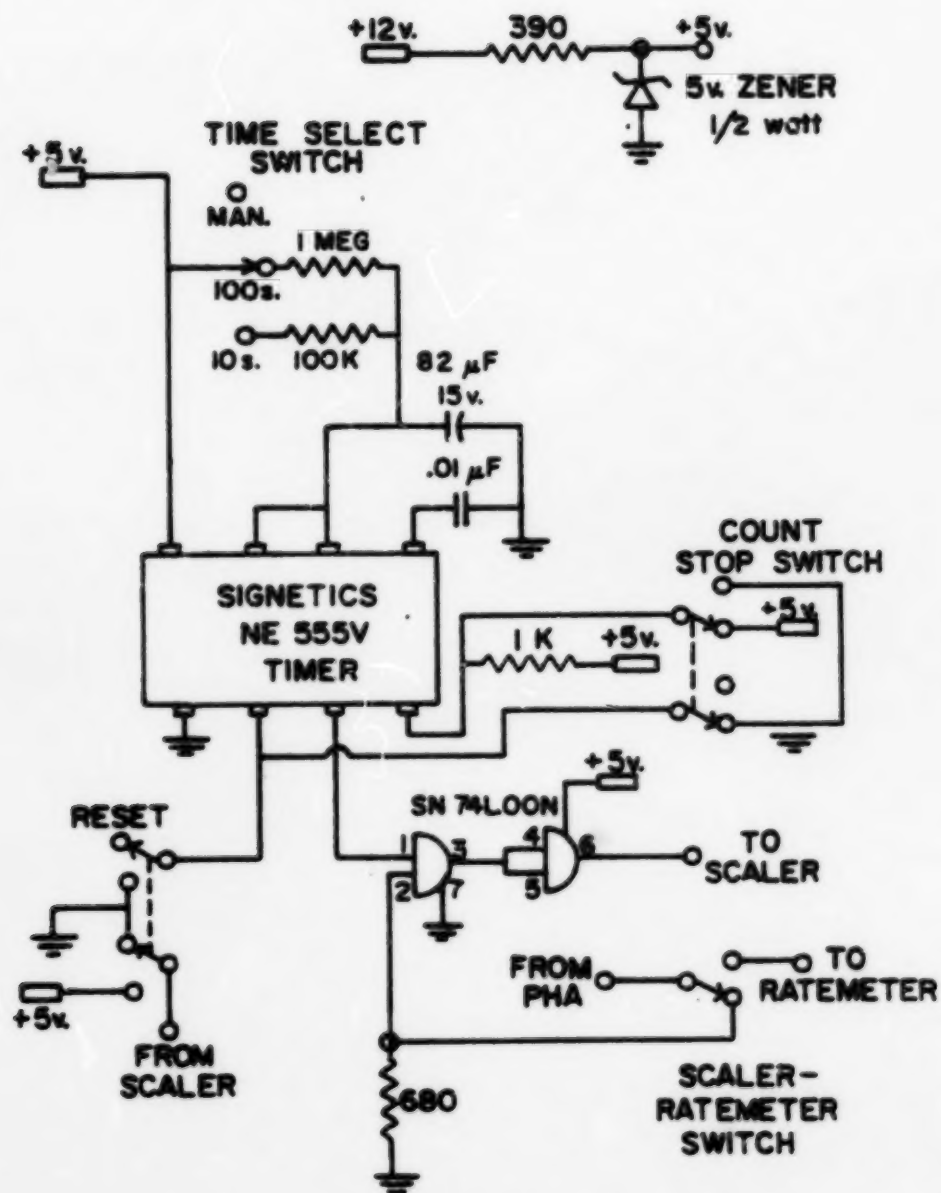




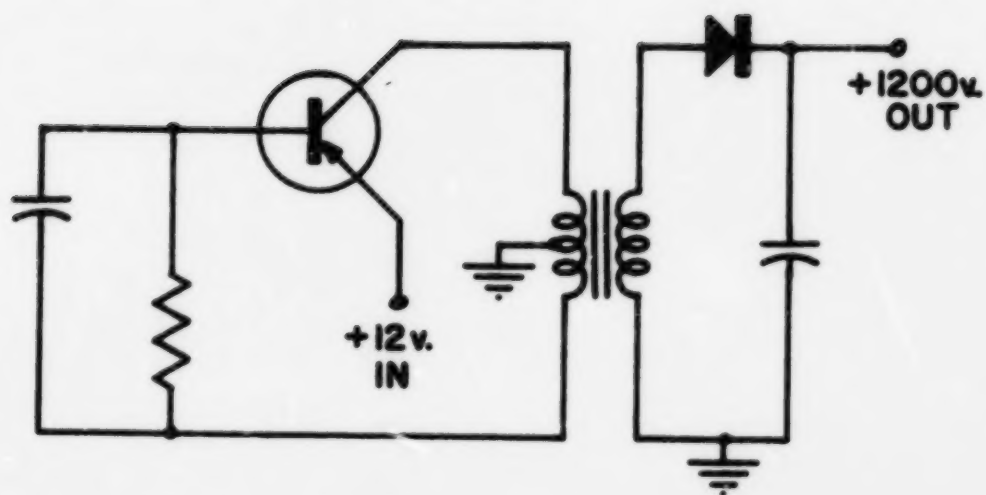
Circuit 3 Ratemeter (Range switch resistor values need to be determined at time of installation; a pulser is used to feed known frequencies and the resistor values adjusted to provide appropriate deflection on the meter.)



Circuit 4 Scaler



Circuit 5 Timer



Circuit 6 Typical detector high-voltage supply